#### OFFICE OF NAVAL RESEARCH



## FINAL REPORT

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## Solid Electrolytes for Multivalent Cations

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93-29103 **MINIMUM** 

## a. Brief Description of Project

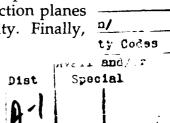
The ß and ß" aluminas are well appreciated as a unique family of materials with remarkable structural, transport and optical properties. Their rich ion exchange chemistry makes it possible to chemically 'tune' the mobile ion composition so that specific properties are developed within the  $\mathfrak B$ " alumina framework. As ionic conductors, the  $\mathfrak B$ " aluminas are the first solids to exhibit high conductivity for divalent and trivalent cations, while the transition-metal and lanthanide-containing  $\mathfrak B$ " aluminas exhibit some exceptional optical properties. The present research program builds upon the structure/property relationships that we have established for the multivalent  $\mathfrak B$ " aluminas.

The overall objective of the research is to utilize the ß and ß" alumina family of materials as a model system in which to design and synthesize compounds with pre-determined properties. This family of materials (which includes aluminates, ferrites and gallates) is unique because a single structure type is maintained as one alters not only the chemical nature of the mobile ions but also the chemical nature of the framework. The program involves interrelated activities including crystal growth, chemical synthesis and studies of structure, ion transport, hydration and optical properties. An important focus of the project is to closely couple model experiments on structure, ion transport and optical behavior with molecular dynamics (MD) simulations.

## b. Significant Results

This program has carried out wide-ranging studie. of the preparation, chemical properties, electrochemical and optical characteristics, and structural aspects of the multivalent beta" aluminas, the first family of solid electrolytes in which divalent and trivalent cations are mobile. Potential applications of these materials that have been explored are in solid state lasers, phosphors, sensors, and batteries.

Some of the most intriguing results of this work have come from molecular dynamics simulations of the structure and dynamics of ion motion in both ß and ß" alumina. Simulations of the sodium containing forms as well as mixed sodium-cadmium ß" alumina have provided profound insight into the mechanisms by which ions move in the structures on the unit cell level. Simulations have focused on understanding the influence of interstitial oxygen ions on conductivity in sodium ß alumina. These have clearly shown that the interstitial oxygen ions can serve as local traps for mobile sodium ions and that the introduction of divalent cations to the structure releases sodium ions from these traps and increases the effective charge carrier concentration in the structure. Simulations of sodium ß" alumina have begun to reveal the influence of magnesium ions in the structure on ion arrangement and conductivity in the conduction planes and also have shown how local ion-vacancy ordering leads to conduction planes which are actually dynamic mosaics of ion motion and immobility. Finally,



simulations of mixed sodium-barium ß" alumina have 'predicted' with remarkable accuracy the variation of ion transport observed experimentally as a function of composition. The simulations, however, provide detailed insight into the local structures responsible for the variations.

Taken together, these simulation studies have advanced considerably our understanding of this particular set of compounds as well as the techniques used to simulate and predict the characteristics of ion motion in solid electrolytes in general. The original goals of this work were to use simulation to predict the properties of experimentally unknown compositions, and progress towards this goal, at least in one small set of real and hypothetical materials, has been considerable indeed.

In addition, another fascinating study has been completed of the refractive index of ion exchanged  $\beta''$  aluminas. The unique ability to change the composition of the compound without changing its structure type has made it possible to compare experimental results with calculations derived from theoretical treatments. Measurements of the ordinary and extraordinary refractive indices were readily fit to the Clausius-Mossotti relation which is based on highly ionic compounds. These results confirm that cation-oxygen bonds for conduction plane ions are extremely ionic; with the c-direction exhibiting greater ionicity than within the ab plane. Similar conclusions were reached in the spectroscopy of Eu(III)  $\beta''$  alumina. In related work on  $\beta$ -alumina, a mixed Na(I)-Ag(I) composition was able to attain an iso-index point for wavelengths in the visible.

Spectroscopy studies of infrared emitting ions Ho(III) and Er(III) were completed and their prospects as laser materials were evaluated. Both ions exhibited extremely strong hypersensitive transitions. This characteristic of the  $\mathfrak{G}''$  alumina structure arises from the ability of lanthanide ions to be slightly displaced from their equilibrium positions in the conduction plane. Stimulated emission cross sections for Ho (at 2.1  $\mu$ m) and Er (at 1.6 $\mu$ m) gave values comparable to those for typical laser hosts and indicates that laser oscillation can be produced for these ions in the  $\mathfrak{G}''$  alumina host lattice. The high quantum efficiency ( $\approx$ 100%) for Er(III) is particularly attractive for laser action.

Research on the synthesis and properties of ß and ß" aluminogallates emphasized studies of the defect chemistry of the grown single crystals of the sodium compounds. The non-stoichiometry in the ß" aluminogallates was attributed to Mg(II) substitution for Al(III) in the spinel block. The composition corresponds to the formula  $Na_{1+x}Al_x(Al_{1-y}Ga_y)1_{1-y}O_{17}$  with x in the range of 0.67. The defect chemistry of the ß-aluminogallates is less clearly established. The excess sodium content is initially in the range of 0.7, however, simple chemical washing in NaNO3 lowers this value to 0.3. The origin of this behavior and its effect on conductivity are being investigated. NMR results have shown that Ga(III) substitutes preferentially for Al(III) in tetrahedral sites in the spinel block.

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#### g. Patents Filed

none

#### h. Patents Granted

none